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Publication date:
2013

Document Version
Peer reviewed version

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Citation (APA):
Engelbrekt, C., Ulstrup, J., & Zhang, J. (2013). *Dynamic Approach to the Study of Green Nanoparticle Formation*. Abstract from 14th International Symposium on Electroanalytical Chemistry, Changchun, China.

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Dynamic approach to the study of green nanoparticle formation

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Many challenges in modern energy conversion and storage reduce to the efficiency and optimal utilization of catalysts, both determined by the shape, size and composition of the catalyst nanoparticles. Precise control of these parameters of metallic nanoparticles is therefore crucial. A number of synthetic protocols that provide size and shape control have been developed but fundamental investigation of the mechanisms controlling the nucleation and growth of nanoparticles is also gaining increasing focus. X-rays are traditionally utilized to follow oxidation states and cluster size during gold nanoparticle formation. Especially synchrotron sources are employed in order to achieve high enough signal at sampling rates sufficient for the fast reactions. This approach has certain limitations. One is that the availability of these powerful facilities is limited. A second one is that so far slight changes in the syntheses protocols can alter drastically the mechanisms derived. Direct transfer of mechanistic insight from one system to another is therefore challenging.

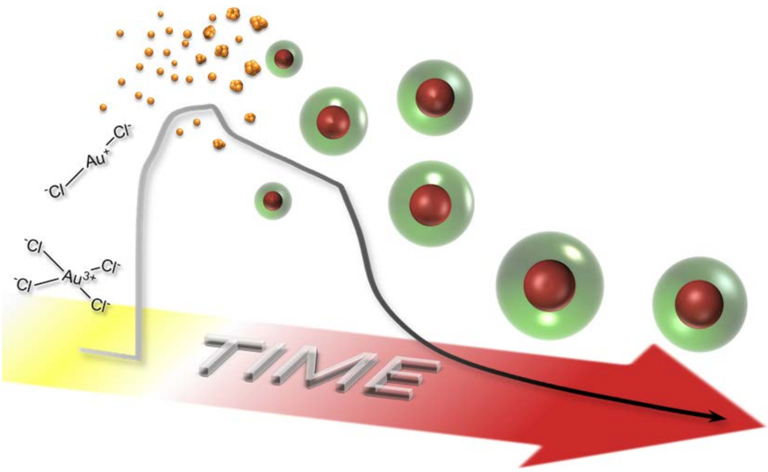

Figure 1. Our platform reveals distinct phases as the precursor goes through several intermediate stages before stable gold nanoparticles are formed. The sketch shows the development of the solution electrochemical potential during formation.

We present here an attempt to tackle these obstacles using a complementary platform for following directly nanoparticle formation mechanisms, Figure 1. The platform should be widely accessible to allow broad collection of data, ultimately enabling a deeper understanding of the underlying mechanisms. Other information about the synthesis mixtures besides the metal precursor will strengthen the understanding of the processes.

Our platform is comprised of well-known and readily available electrochemical and optical techniques. The nanoparticle formation is monitored by following in real time the solution potential, pH, conductivity, turbidity and ultraviolet-visible light absorption supported by transmission electron microscopy and nanoparticle tracking analysis. A "green" saccharide-based approach to metallic nanostructure synthesis (SAMENS) has, further been discovered for both gold and platinum nanoparticles, with mild chemicals and synthesis conditions[1,2], and constitutes the core of our platform. Our standard gold nanoparticle synthesis is chosen as a model system due to its suitable reaction time at room temperature (≈ 15 min).

The dynamic approach shows clearly several distinct phases of chemical reaction, nucleation and growth[3]. Examples indicated by the letters a-g are shown in Fig.2. The different techniques enable us to follow the two-step reduction of the precursor, the release of ligands, the appearance of solid particles and the adsorption of molecules on the fresh nanoparticle surface.

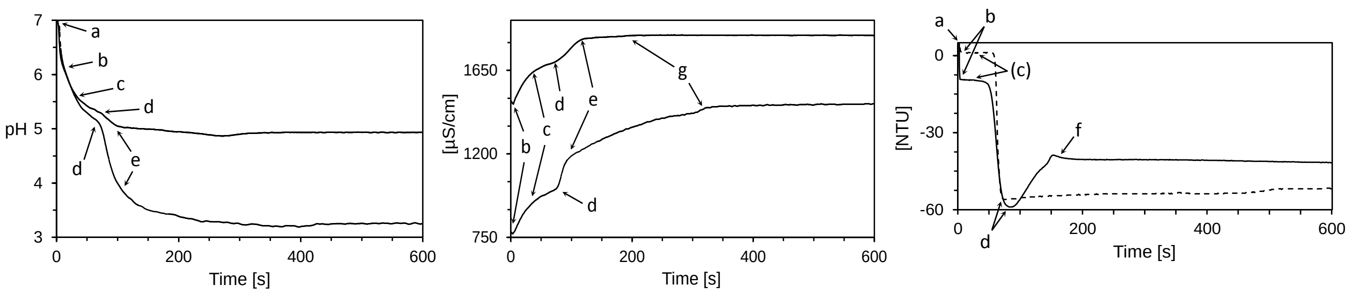

Figure 2. Distinct phases in the gold nanoparticle formation process appear, for example in the time evolution of pH (left), conductivity (middle) and turbidity (right). Solid and dotted lines represent the standard synthesis (Starch, glucose, buffer with addition of gold precursor) and a reference synthesis (buffered aqueous solution adding gold precursor), respectively.

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Acknowledgment

Financial support from the Lundbeck (R45-A3878) and Department of Chemistry for co-financing Ph.D to C. E. is acknowledged. This contribution is integrated in the EU programme ELECTRONANOMAT (IRSES project no. 318990).